

critical field is applied (Meyer & Tagland, 1956). No definite evidence for magnetic ordering in the compound AuMn_3 has so far been found (Meyer, 1959).

Powder photographs of the material, after annealing at 500°C for 24 hours, were initially indexed on a tetragonal cell with $a=3.328 \text{ \AA}$ and $C=8.539 \text{ \AA}$. This indexing did, however, leave a few weak unexplained lines and density determinations gave 1.48 formula units per unit cell. A larger cell with a increased by a factor of $\sqrt{2}$ allowed all the observed lines to be indexed and gave 2.96 formula units per unit cell. The parameters of the larger cell were: $a=4.706$, $C=8.539 \text{ \AA}$. Most of the observed lines satisfied the face-centred selection rule and lines with $l=3n$ were strong.

The dimensions of the cell and its relationship to Au_2Mn suggested that the structure must be of the form:

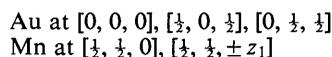


Table 1. $\sin^2 \theta$ values, using copper $K\alpha$ radiation, and observed and calculated structure factors for AuMn_3 assuming $z = \frac{1}{3}$.

Reflexion	$(\sin^2 \theta)_o$	$(\sin^2 \theta)_c$	F_o	F_c
002	0.0324	0.0325	125	153
111	0.0615	0.0616	147	150
200	0.1068	0.1070	364	357
113	0.1267	0.1268	325	347
004	—	0.1302	—	138
210	—	0.1338	—	46
202	0.1393	0.1396	130	138
211	—	0.1419	—	45
220	0.2139	0.2141	304	310
204	0.2368	0.2372	123	127
222	0.2470	0.2467	118	127
115	0.2569	0.2570	137	126
311	0.2761	0.2758	139	125
006	0.2927	0.2929	288	285
313	0.3405	0.3409	290	271
224	—	0.3443	—	120
206	0.3998	0.4000	260	259
400	0.4279	0.4282	256	256
117	0.4529	0.4523	108	113
315	0.4705	0.4711	131	113
226	0.5065	0.5071	221	241
420	0.5349	0.5353	208	237
333	0.5543	0.5550	227	233
404	0.5580	0.5584	—	110
317	0.6654	0.6664	122	105
424	—	0.6655	—	105

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Crystal data (I) for some bile acid derivatives*. By DORITA A. NORTON and BARBARA HANER, *Roswell Park Memorial Institute, Buffalo, New York 14203, U.S.A.*

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Single-crystal data for thirteen bile acid derivatives were collected on a General Electric XRD-5 X-ray diffraction unit equipped with a single-crystal orienter. Reciprocal lattice measurements were made using $\text{Cu } K\alpha$ radiation. Space groups were determined by systematic absences and considerations of optical activity. Densities were determined by flotation and used to calculate the number of molecules

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Mn at $[0, 0, \pm z_2]$, $[\frac{1}{2}, 0, \frac{1}{2} \pm z_2]$, $[0, \frac{1}{2}, \frac{1}{2} \pm z_2]$
where z_1 and z_2 are $\sim \frac{1}{3}$.

This structure would be face centred if the Mn atom at $[\frac{1}{2}, \frac{1}{2}, 0]$ were replaced by gold.

The line intensities were determined by measuring the area of the traces from a Philips powder diffractometer. Anomalies, due to preferred orientation in the closely packed powder, were eliminated by dilution with gum tragacanth. Observed and calculated structure factors for $z_1 = z_2 = \frac{1}{3}$ are shown in Table 1. Where reflexions overlapped, F_o for the strongest line was estimated by subtracting the calculated intensity of the weaker lines from the total observed intensity. Copper radiation was used and all observed spectra up to 313 are recorded in the table. Selected lines at higher angles were measured to fix z within as close limits as possible. From the results it is estimated that assuming $z_1 = z_2$, the best value of z is 0.333 ± 0.007 . The results are not sufficiently precise to make a distinction between z_1 and z_2 possible.

The analysis shows the close similarity between the structures of Au_2Mn and AuMn_3 : if in AuMn_3 the manganese atom at $[\frac{1}{2}, \frac{1}{2}, 0]$ were replaced by gold the structure would be similar to that of Au_2Mn but with manganese at the gold sites and gold at the manganese sites. There are, however, important differences in the environment of the manganese atoms, which would carry the magnetic moment in a magnetically ordered structure. In Au_2Mn the shortest Mn–Mn distance, along $[100]$ and $[010]$ directions, is 3.36 \AA . In AuMn_3 the shortest Mn–Mn distances are the nearest-neighbour distances, in the approximately close-packed (100) and (010) planes, of 2.75 or 2.85 \AA .

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References

- HALL, E. O. & ROYAN, J. (1959). *Acta Cryst.* **12**, 607.
HERPIN, A., MERIEL, P. & MEYER, A. J. P. (1958). *C. R. Acad. Sci., Paris*, **246**, 3170.
MEYER, A. J. P. (1959). *J. Phys. Radium*, **20**, 430.
MEYER, A. J. P. & TAGLAND, P. (1956). *J. Phys. Radium*, **16**, 341.
RAUB, E., ZWICKER, U. & BAUR, H. (1953). *Z. Metallk.* **44**, 312.

per unit cell. All of the compounds studied were crystallized from solution, and, in most cases, evaporation at room temperature gave a yield of good single crystals. The calculated molecular weights of compounds 2, 5, 8, 11, and 13 show that solvent of crystallization is present and that this solvent is water in 5, 11, and 13. The unusually high discrepancy between the observed and calculated densities of compound 9 probably results from the inaccuracy of the density determination of the crystals, which were small, extremely thin laths. The crystal data are given in Table 1. No further work on these compounds is contemplated.

Table 1. Crystal data for thirteen bile acid derivatives

	1	2	3	4	5
Formula	$C_{22}H_{33}O_3$	$C_{24}H_{40}O_4 \cdot C_2H_5OH$	$C_{24}H_{40}O_4$	$C_{24}H_{40}O_4$	$C_{24}H_{40}O_5 \cdot 4H_2O$
Mol. Wt.	345.51	438.63	392.56	392.56	480.64
ρ meas. (g.cm ⁻³)	1.149	1.160	1.154	1.174	1.156
ρ calc. (g.cm ⁻³)	1.154	1.152	1.170	1.185	1.153
Space group	$P2_12_12_1$	$P2_12_12_1$	$P2_12_12_1$	$P2_12_12_1$	$P2_1$
Z	4	4	4	8	2
$a(\text{\AA})^*$	11.794	13.589	11.577	13.302	14.043
$b(\text{\AA})^*$	28.039	25.765	29.924	26.737	7.849
$c(\text{\AA})^*$	6.019	7.225	6.433	12.377	13.697
α	—	—	—	—	—
β	—	—	—	—	113.5°
γ	—	—	—	—	—
$V(\text{\AA}^3)$	1990	2530	2229	4402	1384
Solvent	Methanol	Ethanol—acetone	80 % Ethanol	80 % Ethanol—ethyl acetate	Ethyl acetate

* ± 0.008

- 22,23-Bisnor-5-cholenic acid-3 β -ol
- 5 β -Cholan-3 α ,12 α -diol . C_2H_5OH (deoxycholic acid . C_2H_5OH)
- 5 β -Cholan-3 α ,6 α -diol
- 5 β -Cholan-3 α ,7 β -diol
- 5 β -Cholan-3 α ,7 α ,12 α -triol . $4H_2O$ (cholic acid hydrate)

	6	7	8	9	10
Formula	$C_{24}H_{42}O$	$C_{24}H_{39}O_5Na$	$C_{25}H_{42}O_5 \cdot C_2H_5OH$	$C_{25}H_{42}O_3$	$C_{27}H_{42}O_5$
Mol. Wt.	346.60	430.57	468.68	390.61	446.63
ρ meas. (g.cm ⁻³)	1.114	1.167	1.163	1.132	1.146
ρ calc. (g.cm ⁻³)	1.098	1.191	1.167	1.217	1.170
Space group	$C2$	$P2_1$	$C2$	$P2_12_12_1$	$P2_12_12_1$
Z	12	2	4	4	8
$a(\text{\AA})^*$	40.689	12.593	25.489	11.337	11.448
$b(\text{\AA})^*$	6.907	8.215	8.011	26.796	59.335
$c(\text{\AA})^*$	29.046	12.196	15.337	7.013	7.468
α	—	—	—	—	—
β	129.61°	107.86°	121.59°	—	—
γ	—	—	—	—	—
$V(\text{\AA}^3)$	6289	1201	2668	2130	5073
Solvent	Acetone	Acetone	Ethanol	Ethanol	Acetone

* ± 0.008

- 5 β -Cholan-24-ol
- 5 β -Cholan-3 α ,7 α ,12 α -triol sodium salt (cholic acid sodium salt)
- 5 β -Cholan-3 α ,7 α ,12 α -triol methyl ester . C_2H_5OH
- 5 β -Cholan-3 α -ol methyl ester (lithocholic acid methyl ester)
- 5 β -Cholan-3 α -ol-12-one acetate methyl ester

	11	12	13
Formula	$C_{27}H_{44}O_4$	$C_{28}H_{42}O_7$	$C_{29}H_{44}O_7 \cdot \frac{1}{2}H_2O$
Mol. Wt.	432.65	490.64	513.68
ρ meas. (g.cm ⁻³)	1.168	1.123	1.197
ρ calc. (g.cm ⁻³)	1.144	1.141	1.192
Space group	$P2_1$	$P1$	$C2$
Z	2	2	12
$a(\text{\AA})^*$	14.989	14.557	47.900
$b(\text{\AA})^*$	7.740	16.671	7.995
$c(\text{\AA})^*$	11.355	6.343	22.922
α	—	93.47°	—
β	107.43°	98.18°	102.06°
γ	—	106.51°	—
$V(\text{\AA}^3)$	1258	1429	8584
Solvent	Ethanol	Ethanol	Ethanol—acetone

* ± 0.008

- 5 β -Cholan-3 α -ol-acetate methyl ester .
- 5 β -Cholan-3 α ,7 α -diol-12-one diacetate
- 5 β -Cholan-3 α ,7 α ,12 α -triol 3,7-diacetate methyl ester . $\frac{1}{2}H_2O$